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Promotion of selectivity through organised media: Fries rearrangement of calix (n) arene esters (n = 4,6,8)†

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Fries migration of various calix(n)arene esters (n = 4,6,8) under the influence of different solvents (benzene, nitrobenzene, chlorobenzene, carbon disulphide, toluene, xylene, mesitylene) and Lewis acid catalysts (aluminium chloride, ferric chloride and boron trifluoride etherate) has been examined. It appears that the best conditions for the conversion of calix(n)arene esters to *p*-acyl calix(n)arenes involve the use of aluminium chloride as the Lewis acid and carbon disulphide or benzene as solvent. The use of toluene or mesitylene as a solvent leads to regioselective formation of their acyl derivatives probably through the intermediacy of a host guest complex of the solvent with calix(n)arenes.

The use of organised media for the promotion of selectivity in organic transformations is a subject of current interest[1]. Though inorganic constrained systems and media have been extensively utilized in directing simple organic transformations[2-7], the use of organic host guest complexes for this purpose has been studied to a limited extent[8-11]. As a part of our programme to investigate the use of organised media for selective organic transformations, we have carried out the Fries rearrangement on calix(n)arene esters (n = 4,6,8), with a view to elucidate the principles involved in the execution of intended goals of stereoselective functionalizations through organized media. Calix(n)arenes[12,13] (n=4,6,8) are a well recognised class of metacyclophanes with an enormous potential to develop new molecular receptors for ionic[14,15] and molecular recognition[16-19]. Their potential as enzyme mimics was suggested in 1983 but so far only a few reports[20-24] have appeared which evince their catalytic powers or their use as organised media albeit with efficiencies that are much less than the enzyme reactions thereby providing impetus to design new calixarene based biomimetic systems. Fries migration of calixarene esters is one of the important reactions that would provide substituted calixarene ketones for further manipulation. Since the *ortho*-positions of parent calix(n)arenes are already involved in their σ frame work through methylene bridge, Fries reaction is expected to yield only the *para*-acylcalix(n)arenes which should have profound practical utility. Literature survey indicates that Fries migration of calix(n)arene esters has been studied by Shinkai *et al.*[25] and No *et al.*[26-29]. While No and coworkers have studied the preparation of acetylated dimethyl ethers and their subsequent conversion to the *p*-acylderivatives of calix(n)arenes[26-29], Shinkai *et al.* have determined that acylated calix(6)arenes undergo Fries migration in 29-34% yields[25]. Under similar conditions they have also hinted that acetylcalix(6)arenes do not undergo Fries migration and no reasons could be assigned to the observation. Besides, the earlier authors have neither studied the behaviour of calix(4)arene esters nor that of calix(8)arene esters. In this paper, we describe the details of our work on the Fries migration of calix(n)arene esters (n=4,6,8) in the presence of various solvents (benzene, nitrobenzene, chlorobenzene, carbon disulphide, toluene, xylene, mesitylene) and Lewis acids (aluminium chloride, ferric chloride and boron trifluoride etherate) to yield the corresponding *p*-acylcalix(n)arenes and provide plausible explanation for the results obtained. The study points out to the fact that calix(n)arenes can be used as the organised media for a variety of

†Dedicated to Prof. Waldemar Adam on his 60th birthday

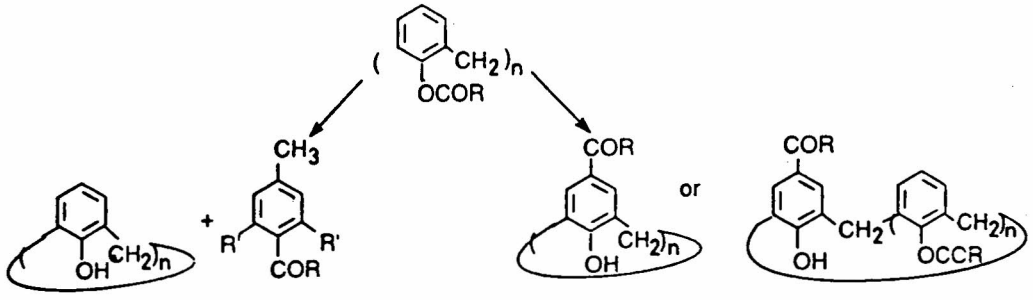
reactions. It has been observed that the Fries migration on calixarene esters is successful only when carried out in carbon disulphide or benzene, while it is not favoured in toluene, xylene and mesitylene. These solvents when used, themselves undergo regioselective acylation. The use of nitrobenzene as a solvent however does lead to *p*-acylated calixarenes but the removal of nitrobenzene from products is difficult probably due to the formation of strong association complexes between the acylated calix(n)arenes and nitrobenzene. The best Lewis acid for Fries migration of calix(n)arene esters has been found to be the anhydrous aluminium chloride while boron trifluoride and ferric chloride are not as effective.

Results and Discussion

Fries migration of 25, 26, 27, 28-tetraacetoxycalix(4)arene **1a** on treatment with aluminium chloride in nitrobenzene at 50°C gave 5, 11, 17, 23-tetracetyl-25, 26, 27, 28-tetrahydroxycalix(4)arene **4a** in 50% yield (Scheme I, Table I). As it was difficult to remove nitrobenzene from the reaction mixture completely, change in reaction conditions involving chlorobenzene as the solvent was attempted. Consequently, Fries migration of 37,

Table I—Fries rearrangement of calix(n)arene esters (n =4, 6. 8) in different solvents in the presence of anhydrous $AlCl_3$ as catalyst

Substrate	Temperature	Product	Yield %
1a	Nitrobenzene 50°C	**40a**[30]	50
1a	Benzene 70°C	**4a**	35
1a	Carbon disulphide R T/72hr	**4a**	30
1b	R T/72hr	**4b**	30
2a	R T/72hr	**5a**	28
2b	R T/72hr	**5b**	20
2a	Chlorobenzene 50°C	Black resin	—
2b	50°C	Black resin	—
2c	50°C	**5c**[34]	29
2d	50°C	**5d**[34]	34
1a	Toluene 70°C	**7a**	40
1b	70°C	**7b**	45
2a	70°C	**7a**	40
2b	70°C	**7b**	45
1a	Mesitylene 70°C	**1a**	—
1a	*m*-Xylene 70°C	**4c**	45
3a	Mesitylene R T/72h	**7c**	20



8 n = 4 7a R = CH_3, R' = H
9 n = 6 7b R = C_6H_5, R' = H
10 n = 8 7c R = CH_3, R' = CH_3

1a n = 4 R = CH_3
1b n = 4 R = C_6H_5
2a n = 6 R = CH_3
2b n = 6 R = C_6H_5
2c n = 6 R = $-(CH_2)_4CH_3$
3a n = 8 R = CH_3
3b n = 8 R = C_6H_5
2d n = 6 R = $CH\text{-}CH_2CH_3$ (with CH_3 branch)

4a n = 4 R = CH_3
4b n = 4 R = C_6H_5
5a n = 6 R = CH_3
5b n = 6 R = C_6H_5
5c n = 6 R = $-(CH_2)_4CH_3$
6a n = 8 R = CH_3
6b n = 8 R = C_6H_5
5d n = 6 R = $CH\text{-}CH_2CH_3$ (CH_3 branch)

4c n = 3 R = CH_3

Scheme I

38, 39, 40, 41, 42-hexakis (hexanoyloxy) calix(6)arene **2c** and 37, 38, 39, 40, 41, 42-hexakis (2-methylbutanoyloxy)calix(6)arene **2d** in chlorobenzene using aluminium chloride as a catalyst was monitored, but the yield of the corresponding calixarene ketone[25] obtained was low. Analogously, the rearrangement of 25, 26, 27, 28-tetraacetoxycalix(4)arene **1a** and 37, 38, 39, 40, 41, 42-hexaacetoxycalix(6)arene **2a** under similar reaction conditons was not clean. The use of more drastic conditions (high temperature or increase in quantity of aluminium chloride) led to the formation of a black resinous mass which was insoluble in most common solvents. The change of Lewis acid from aluminium chloride to boron trifluoride in dimethyl ether did not provide the expected product. Modifications of the reaction conditions by replacing chlorobenzene with carbon disulphide/benzene in the above rearrangement reaction as well as calixarene acetates and benzoates resulted in the isolation of calixarene ketones in 26-30% yields. When the same reaction was repeated in toluene at 70°C, the reaction did not yield the calixarene ketones but provided the products which indicated the acylation of the solvent thereby revealing the intermolecular nature of the rearrangement. For instance, 25, 26, 27, 28-tetraacetoxycalix(4)arene **1a** and 37, 38, 39, 40, 41, 42-hexaacetoxycalix(6)arene **2a** on treatment with $AlCl_3$ in toluene at 0°C, did not yield any *para*-acetylated calix(n)arenes (n=4,6). On increasing the temperature to ~70°C, the reaction mixture showed the appearance of four spots on TLC which after separation through column chromatography gave three compounds. These compounds when subjected to chemical and spectral analyses were proved to be *p*-methylacetophenone **7a**, 25, 26, 27, 28-tetrahydroxycalix(4)arene **8** and 37, 38, 39, 40, 41, 42- hexahydroxycalix(6)arene **9** respectively.

Similar behaviour was observed in the case of 25, 26, 27, 28-tetrabenzoyloxycalix(4)arene **1b** and 37, 38, 39, 40, 41, 42-hexabenzoyloxycalix(6) arene **2b**, when *p*-methylbenzophenone **7b** was formed along with 25, 26, 27, 28-tetrahydroxycalix(4)arene **8** and 37, 38, 39,40, 41, 42-hexahydroxycalix(6)arene **9** respectively. Fries migration of 49, 50, 51, 52, 53, 54, 55, 56-octaacetyloxycalix(8)arene **3a** in toluene at ~70°C failed to give either calixarene ketones or *p*-methylacetophenone **7a**. Altering the solvent from toluene to *m*-xylene at 70°C, in the case of 25, 26, 27, 28-tetracetyloxycalix(4)arene **1a**, led to the formation of a partially Fries migrated product, 5-acetyl-25, 26, 27-triacetoxy-28-hydroxycalix(4)-arene **4c**. On the other hand, Fries migration of 49, 50, 51, 52, 53, 54, 55, 56-octaacetoxycalix(8)arene **3a** in mesitylene at room temperature resulted in the formation of 2,4,6-trimethylacetophenone in 20% yield while the use of mesitylene as the solvent evinced no change in the reaction of 25, 26, 27, 28-tetracetoxycalix(4)arene **1a** and 37, 38, 39, 40, 41, 42- hexaacetoxycalix(6) arene **2a**.

The nature of the rearrangement of calixarene esters was examined by carrying out the reaction in different solvents. Since toluene was observed to be acylated in the Fries migration of calix(n)arene (n=4,6) esters, the reaction could tentatively be termed as intermolecular. This conclusion however is not unambiguous as the absence of acylation of other solvents (e.g., nitrobenzene and chlorobenzene) could have been due to their lower reactivity than the calix(n)arenes (n=4,6). Subsequently, on repetition of the reaction by using more reactive and higher boiling solvents such as *m*-xylene and mesitylene, the rearrangement did not lead to their acylation in the case of calix(n)arene esters (n = 4,6), while 2,4,6-trimethylacetophenone was obtained in the Fries migration of octaacetylcalix(8)arene in mesitylene. These observations reveal that the reason for acylation of the solvent in the rearrangement of calix(n)arene esters is more complex than the simple intermolecular nature of the Fries rearrangement of calix(n)arene esters.

The formation of *p*-methylacetophenone and *p*-methylbenzophenones from calix(n)arene esters(n = 4,6) under Fries migration conditions could be explained if calix(n)arenes form a host-guest (1:1) endo complex with toluene as reported by Ungaro *et al.*[24-32] Since no *o*-methylacetophenones and *o*-methylbenzophenones were detected in the reaction mixture, exclusive formation of only *p*-substituted acetophenones and benzophenones indicate that the reaction is regioselective and acyl or benzoyl cation generated by the decomposition of calix(n)arene esters (n=4,6), on treatment with $AlCl_3$, react with the toluene present in the

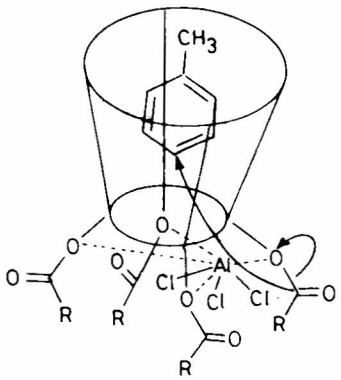


Figure 1

calixarene cavity in the solvent cage itself (Figure 1). Acylation of the solvent was not observed in the rearrangement of calix(4)arene esters when nitrobenzene, *m*-xylene and mesitylene were used as solvents as geometric considerations did not favour their host guest complexation with calix(4)arene esters. This view was supported by a recent X-ray investigation of calix(4)arene derivatives[31,32] as well as the observation that when *m*-xylene was used as solvent, no 2,4-dimethyacetophenones could be detected from the rearrangement reaction of 25,26,27,28-tetraacetoxycalix(4)arene in *m*-xylene. In mesitylene, neither the Fries migration products nor acetylated mesitylene were formed in the Fries rearrangement of calix(4)arene and calix(6)arene esters possibly because mesitylene does not form a host guest complex with calix (n)arenes (n = 4,6), while formation of 2,4,6-trimethylacetophenone in the reaction of octaacetoxycalix(8)arene **3a** in mesitylene could be explained on the basis of larger cavity size of calix(8)arene which can form a host guest complex with mesitylene.

It is significant to note that though apparent yield (26-30%) of Fries migration of calixarene esters seems to be low, it can be considered good when 'n' phenyl esters groups in calixarene are considered individually and the reaction can be useful for obtaining calixarene derivatives with manipulable upper rim substituent. The reaction could also be utilized for affecting the regioselective acylation of the aromatic substrates that form host guest complexes with calix(n)arenes. Further work to use calix(n)arene based molecular receptors for obtaining stereoselective acylation/alkylation is in progress.

Experimental Section

General. Solvents and reagents employed in the study were from Merck, Bombay (AR Grade). Pet. ether refers to the fraction boiling at 60-80°C. Thin layer chromatography (TLC) was performed on silica gel G (Sarabhai Chemicals, Bombay), while column chromatography was carried out over silica gel (60-120 mesh) procured from Qualigens Chemicals Limited, Bombay. NMR spectra were recorded on a Jeol (JNM-FX 100) FT-NMR spectrometer in $CDCl_3$ using tetramethylsilane (TMS) as internal standard. Melting points were taken on an electric melting point apparatus (Toshniwal) and are uncorrected. Molecular masses of calix(n)arenes synthesized were determined by vapour pressure osmometry using Knauer pressure osmometer.

5, 11, 17, 23-Tetra-*tert*-butyl-25, 26, 27, 28-tetrahydroxycalix(4)arene, 5, 11, 17, 23, 29, 35-hexa-tert-butyl- 37, 38, 39, 40, 41, 42-hexahydroxycalix(6)arene and 5, 11, 17, 23, 29, 35, 41, 47-octa-*tert*-butyl-49, 50, 51, 52, 53, 54, 55, 56- octahydroxycalix(8)arene were prepared by the procedures reported in literature[29]. 25,26,27,28-Tetrahydroxycalix(4)arene, 37, 38, 39, 40, 41, 42-hexahydroxycalix(6)arene, 49, 50, 51, 52, 53, 54, 55, 56-octahydroxycalix(8)arene. 25, 26, 27, 28-tetraacetoxycalix(4)arene, 37, 38, 39, 40, 41, 42-hexaacetoxycalix(6)arene and 49, 50, 51, 52, 53, 54, 55, 56-octaacetoxycalix(8)arene. 25, 26, 27, 28-tetrabenzoyloxycalix(4)arene, 37, 38, 39, 40, 41, 42 hexabenzoyloxycalix(6)arene and 49, 50, 51, 52, 53, 54, 55, 56-octabenzoyloxycalix(8)arene were synthesized by slight modifications of the published method[33]. All the known compounds were identified by spectroscopic analysis as well as by their comparison with authentic samples.

General procedure for Fries migration: Fries migration of 1a in nitrobenzene. A mixture of compound **1a** (1.0g, 1.26 mmoles) and anhydrous $AlCl_3$ (1.35g, 0.01 mmole) in nitrobenzene (30 mL) was stirred under a continuous stream of nitrogen for 3hr at room temperature and for additional 6 hr at 60-70°C. The reaction mixture was poured into ice cold water (30mL) containing conc. HCl (3mL). The nitrobenzene layer was separated, washed with water, dried over anhydrous Na_2SO_4 and the solvent removed under

reduced pressure to give a residue which was recrystallized from chloroform as a colourless solid (0.50g, 50% yield), m.p. >300°, Mol. mass: 570 (Calcd. 592). Anal Calcd. for $C_{36}H_{32}O_8$: C, 72.97; H, 5.40. Found: C, 71.92; H, 5.34%; IR(KBr): 1670 (>C=O) cm^{-1}; ^1H NMR ($CDCl_3$): δ7.76 (s, 4H, D_2O exchangeable OH), 6.76 (bs, 8H, Ar*H*), 3.98 (s, 8H, ArCH_2Ar), 2.48(s, 12H, CH_3); ^{13}C NMR ($CDCl_3$): δ 195.5, 156.7, 129.1, 128.9, 30.8, 25.7.

Fries migration of la in carbon disulphide. A mixture of compound **la** (0.5g, 0.63 mmoles) was stirred with $AlCl_3$ (0.62g, 4.64 mmoles) at room temperature in carbon disulphide (25mL) for 72 hr. The reaction mixture was poured into ice cold water (30mL) containing HCl (10%). The carbon disulphide layer was separated and subjected to distillation under reduced pressure to give a white solid which was chromatographed over silica gel using hexane-benzene (1:1) as the eluent. Recrystallization from chloroform -methanol yielded **4a** as a white solid (0.15g, 30% yield), m.p. >300°.

Typical procedure for Fries migration of la in toluene. Compound **1a** (0.51g, 0.64 mmole) was stirred with anhydrous $AlCl_3$ (0.72g, 5.3 mmoles) in toluene (30mL) at ~ 60°C for 3.5 hr. The reaction was quenched by the addition of ice-cold water (50mL) containing HCl (10%) and toluene soluble portion separated, washed with water (2×20 mL), dried over anhydrous Na_2SO_4 and the solvents were removed under pressure. The residue obtained was recrystallized from chloroform-methanol (1:1), to give a white solid which when chromatographed over silica gel yielded two components designated as compound **9** (0.23g, 45% yield, m.p. >300°) and compound **7a** as a semisolid (0.13g, 40% yield); IR(KBr): 1670(>C=O)cm^{-1}; ^1H NMR ($CDCl_3$): δ7.2-7.8 (dd, 4H, Ar*H*).2.5 (s, 3H, OCH_3), 2.4 (s, 3H, ArCH_3 along with decomposed matter and the unreacted starting calix(n)arene derivatives.

Fries migration of 1b in toluene. A mixture of compound **1b** (0.67g, 0.80 mmole) was treated with anhydrous $AlCl_3$ (0.91g, 6.8 mmoles) in toluene (50 mL) and the mixture stirred at 10°C for 3.5 hr. The reaction mixture was worked up in a usual manner to give compounds identified as **9** (0.27g, 40% yield, m.p. >300°) and **7b** (0.26g, 42% yield), m.p. 54-55°; IR (KBr): 1665 (>C=O cm^{-1}); ^1H NMR ($CDCl_3$): δ7.2-7.8 (m, 9H, Ar*H*), 2.4 (s, 3H, $ArCH_3$).

Fries migration of 1b in benzene. A mixture of 25, 26, 27, 28-tetrabenzoyloxycalix(4)arene **1b** (0.47 mmole) was stirred with $AlCl_3$ (0.35g, 0.37 mmole) at 50°C in benzene (30mL) for 48hr. The reaction mixture was poured into ice cold water (25 mL) containing HCl (10%). The benzene layer was separated, dried and subjected to distillation under reduced pressure to yield a white solid which on column chromatography yielded **4b** (35% yield), m.p. 175-178°C; Mol. mass: 820 (Calcd 840). Anal. Calcd. for $C_{56}H_{40}O_8$: C, 80.0; H, 4.76. Found: C, 78.09; H, 4,57%; IR (KBr); 3300(OH), 1662 (>C = O) cm^{-1}; ^1H NMR ($CDCl_3$); δ7.64 (s, 4H, D_2O exchangeable, OH), 7.20-7.47 (m, 28H, Ar*H*), 3.96 (s, 8H, ArCH_2Ar); ^{13}C NMR ($CDCl_3$); δ193.8, 157.1, 137.9, 131.2, 130.8, 129.0, 128.3, 127.9, 30.9.

5,11,17,23,29,35-Hexaacetyl- 37, 38, 39, 40, 41, 42-hexahydroxycalix(6)arene 5a. A mixture of compound **2a** (0.42g, 0.47 mmole) was stirred with $AlCl_3$ (0.75, 5.6 mmoles) in benzene (30 mL) for 50hr at ~ 30°C. The reaction was worked up to give a creamish solid (0.12g, 28% yield), m.p. >300°, Mol. mass; 880(888). Anal Calcd. for $C_{58}H_{48}O_{12}$; C, 72.97; H, 5.40. Found: C, 72.81; H, 5.31%. IR(KBr): 3312(OH), 1660 (>C=O)cm^{-1}; ^1H NMR ($CDCl_3$); δ6.89(s, 12H, Ar*H*), 5.04 (s,12H, ArCH_2Ar). 2.41 (s, 18H, $COCH_3$); ^{13}C NMR ($CDCl_3$); δ195.6, 156.6, 133.1, 128.7, 127.1, 30.4, 25.3.

5, 11, 17, 23, 29, 35-Hexabenzoyl -37, 38, 39, 40, 41, 42-hexahydroxycalix(6)arene 5b. A mixture of compound **2b** (1.09g, 1mmole) and aluminium chloride (1.48g, 0.012mole) in benzene (100 mL) was stirred at ~60°C for 70 hr. The reaction mixture was worked up in a usual manner to give a creamish solid (0.3g, 30% yield), m.p. 258-60°; IR (KBr): 3300(OH), 1662 (>C = O) cm^{-1}; ^1H NMR ($CDCl_3$): δ6.96-7.02 (m, 42H, Ar*H*), 6.16 (s, D_2O exchangeable, 6H, OH), 3.96 (s, 12H, ArCH_2 Ar): ^{13}C NMR ($CDCl_3$): δ193.8, 157.0, 137.8, 131.1, 130.8, 128.7, 128.0, 127.7, 30.8.

5, 11, 17, 23, 29, 35, 41, 47-Octaacetyl-49, 50, 51, 52, 53, 54, 55, 56-octahydroxycalix(8)arene 6a. Compound **3a** (0.61g, 0.51 mmole) was stirred

with $AlCl_3$ (1.2g, 8.9 mmoles) in benzene (50mL) for 50 hr at 30°C. The reaction mixture was worked up to give a white solid (0.17g, 29% yield), m.p. > 300°; Mol. mass: 1170 (Calcd: 1184). Anal. Calcd for C_{17} $H_{64}O_{16}$: C, 72.97: H, 5.40. Found: C, 71.98; H, 5.6%); IR (KBr): 3312(OH), 1660 (> C = O) cm^{-1}; ^1H NMR ($CDCl_3$); δ6.90 (s, 16H, Ar*H*), 4.98 (s, 16H, ArCH_2Ar), 2.43 (s, 24H, $COCH_3$); ^{13}C NMR ($CDCl_3$): δ195.2, 156.2, 128.4, 128.2, 126.4, 29.8, 25.0.

5, 11, 17, 23, 29, 35, 41, 47 - Octabenzoyl - 49, 50, 51, 52, 53, 54, 55, 56 - octahydroxy-calix(8)arene 6b. A mixture of compound **3b** (0.71g, 0.42 mmole) was stirred with $AlCl_3$ (0.90g, 6.7 mmole) at room temperature for 46hr. The reaction was worked up to give a white solid (0.20g, 28% yield), m.p. 213-15°C; Mol. mass: 2480 (Calcd: 2520). Anal Calcd for $C_{112}H_{80}O_{16}$: C, 80.0; H, 4.76. Found: C, 79.5. H, 4.51%. IR (KBr): 3310(OH), 1660 (>C =O)cm^{-1}; ^1H NMR ($CDCl_3$: δ7.40 (s, 8H, OH), 6.92-7.24 (m, 56H, Ar*H*), 3.98 (s, 16H, ArCH_2 Ar); ^{13}C NMR ($CDCl_3$): δ193.7, 157.6, 137.9, 130.8, 128.4, 127.6, 127.4, 127.3, 30.7.

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